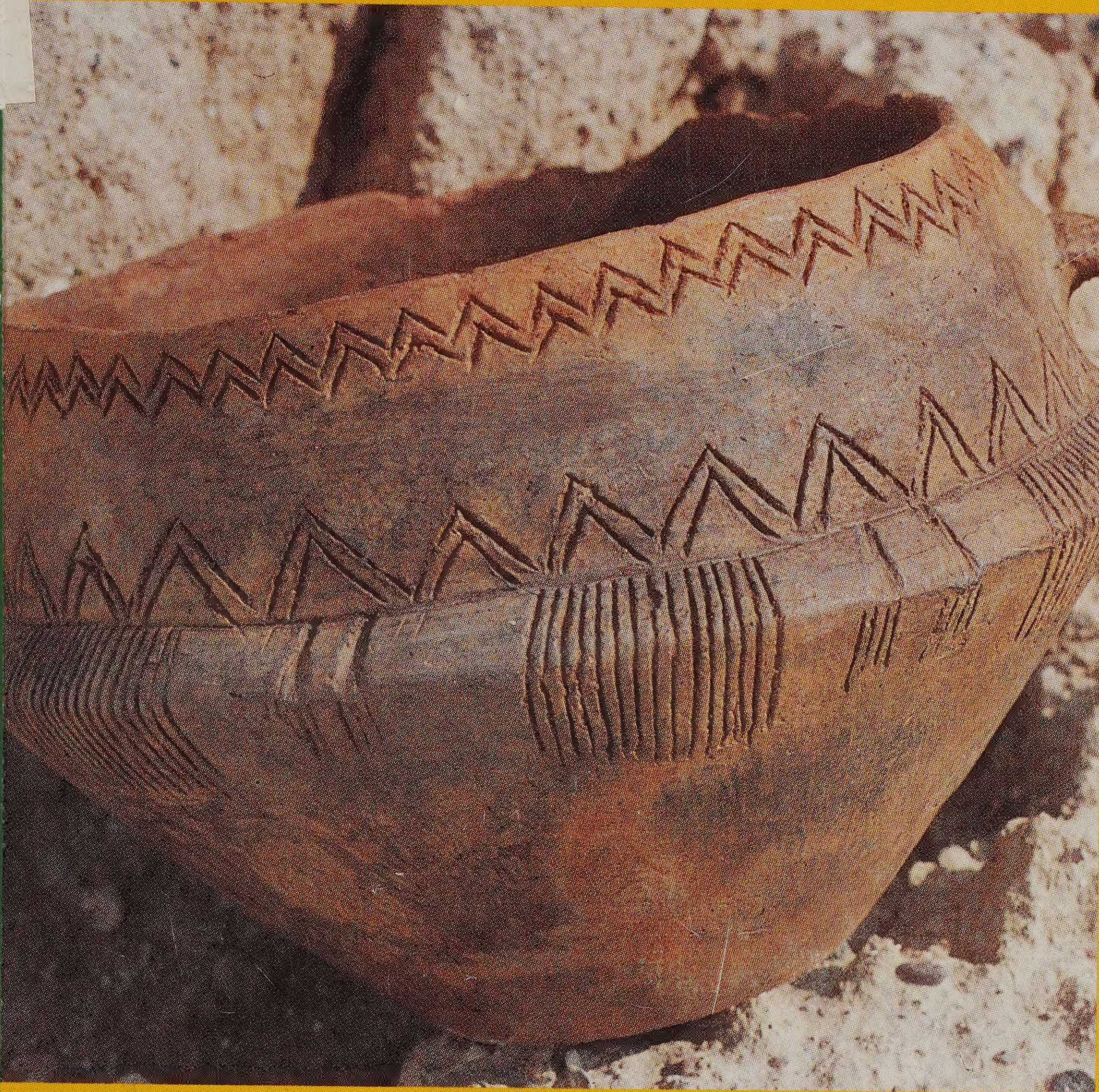

THE NORDIC LABORATORY FOR THERMOLUMINESCENCE DATING

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Cover, front:
Ornamented clay vessel from the Passage Grave Period. Found in June 1971 at Stålmosegård, Vindinge near Roskilde.
Foto: Preben Nielsen.

Cover, back:
Measuring environmental radiation in a sand dune in Mora, Sweden.

May 1988

THE NORDIC LABORATORY FOR THERMOLUMINESCENCE DATING

(Guide for users)

The Nordic Laboratory for TL Dating, established in 1983, is intended primarily for archaeologists and geologists from the Nordic countries, but samples from other countries are also accepted if the laboratory has surplus capacity. The dating capacity is about 200 samples per year. Information on sample taking and submission for dating can be obtained from the laboratory.

A survey is given of the basis for TL dating of archaeological and geological materials, and the dating procedure applied at the Nordic Laboratory is described. Some examples of the application of TL dating are given.

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BASIS FOR TL DATING

TL dating is based primarily on the minerals quartz and feldspar, which are present in most sedimentary deposits and in many materials of archaeological interest, e.g. ceramics and tile. In nature, the two minerals are affected by the radiation to which they are exposed from the omnipresent, naturally occurring radioactive elements uranium, thorium and potassium. The radiation transfers energy to electrons, which enables them to wander through the crystal. Some will be trapped at positions, called traps, where there are imperfections in the crystal, for example a missing negative ion.

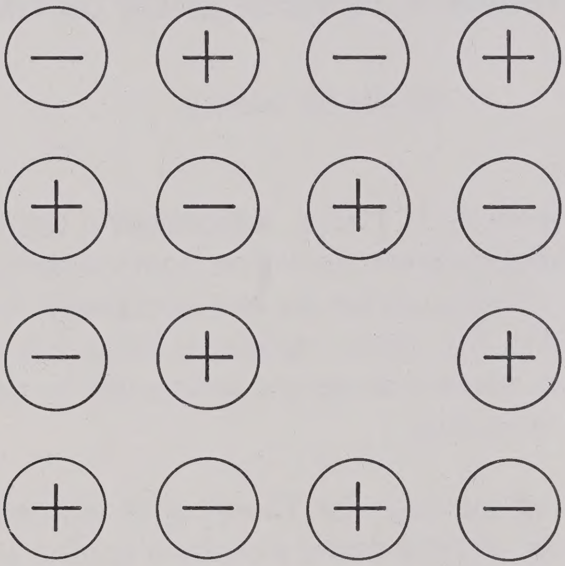


Fig. 1. Crystal lattice. A position where a negative ion is missing will function as an electron trap because there is a surplus of positive charge.

The number of trapped electrons is a measure of the radiation dose absorbed by the material. For minerals in nature, the number will also be related to the time elapsed since the traps were last emptied, because one can assume that the intensity of the natural background radiation has been constant during the period relevant for TL dating, about one million years.

DETERMINATION OF ACCUMULATED DOSE

The radiation dose accumulated in quartz and feldspar can be determined by heating the minerals to a temperature of about 500°C. The heating enables the trapped electrons to escape and return to their ground level. The energy liberated is emitted as a blue/violet light called thermoluminescence (abbreviated TL). The light signal is registered by a light sensitive apparatus. The TL signal from natural samples is compared with that emitted by samples which have been given a know dose of radiation in the laboratory. In this way the unknown dose absorbed by the minerals in nature can be determined.

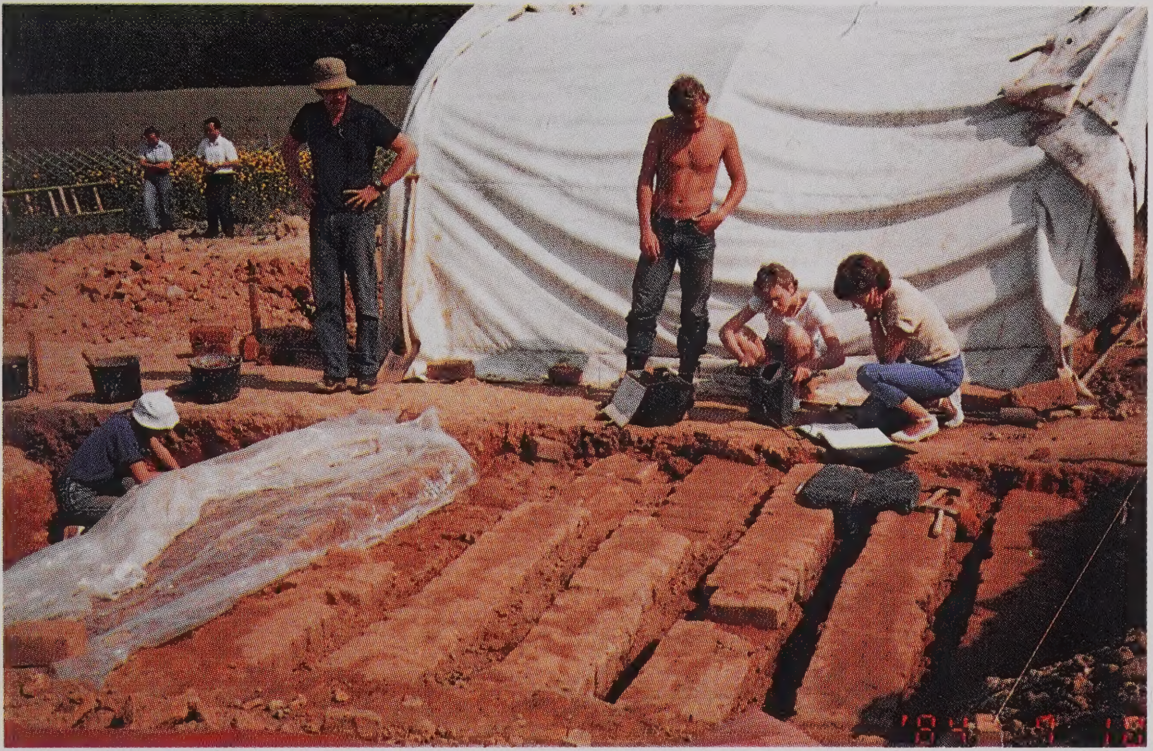


Fig. 2. Taking bricks for dating from a brick kiln at Grønholt near Fredensborg, Denmark. Firing of ceramics and bricks ensures a well-defined zero point for TL dating.

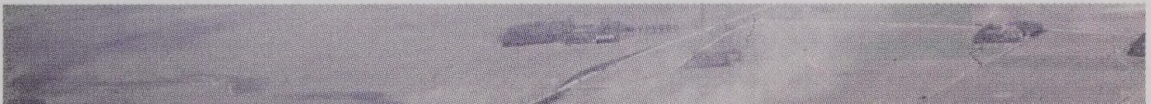
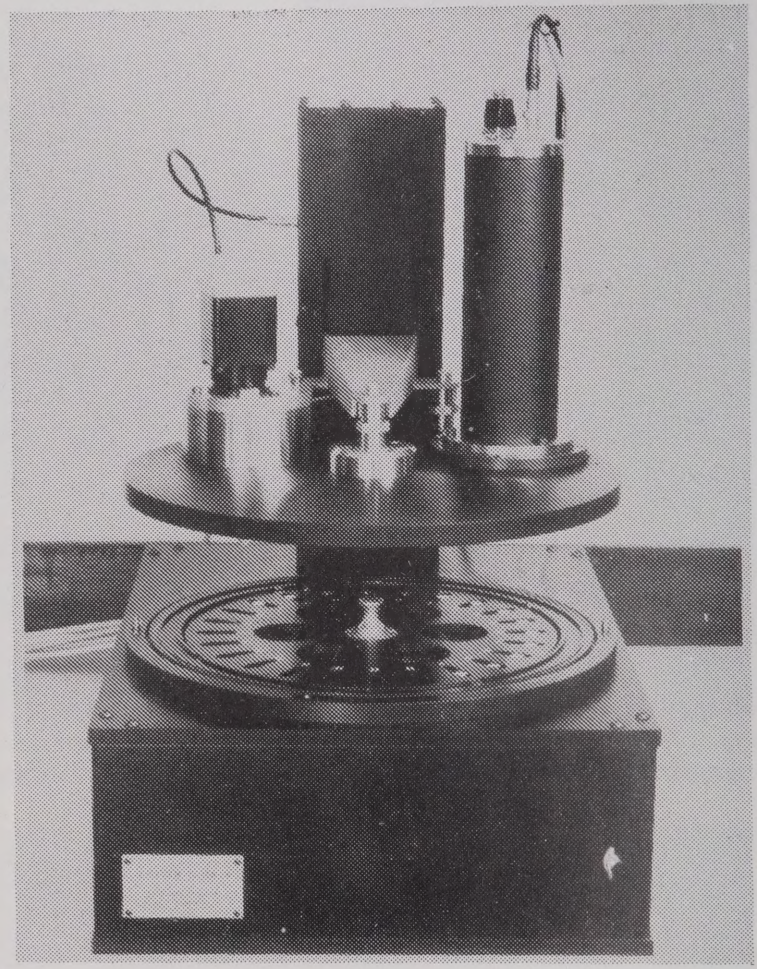


Fig. 3. Sand drift. Exposure of quartz and feldspar to sunlight, during, for example, sand drift, reduces the previously absorbed dose and thereby the TL signal almost to zero. However, zeroing by light is not so efficient as zeroing by heat. Even after prolonged exposure to light a small residual dose remains in the material. But many types of sediments have residual doses small enough to allow TL dating. (Photo: Jens Tyge Møller).

Fig. 4. Automated TL apparatus for measuring TL signals. The sample, placed in a platinum cup, is heated to 500°C by an electric heater element which is brought into contact with the cup. The TL signal is registered by a photomultiplier placed on the lid of the apparatus. The apparatus was developed at Risø.



ZEROING OF THE TL SIGNAL

A necessary requirement for a material to be suitable for dating is that the electron traps were emptied, i.e. the TL signal zeroed, at the time of interest. For archaeological materials, the zeroing was effected by heating during firing or by application of e.g. burnt stones for cooking. This zeroing event is well-defined and effective and corresponds closely to the time of interest for archaeological studies.

The TL signal in sediments has been zeroed as a result of the exposure of the material to light during transport and after deposition, until the deposit in question was covered by subsequent sediments. This zeroing does correspond to the event of interest, but it is less efficient than that resulting from heating; there is always a certain residual dose left in the material. For sediments exposed to light for only a short period, the residual dose can be so high that dating is impossible. The determination of the residual dose present at the time of deposition is one of the most important and difficult tasks in TL dating of sediments.

MATERIAL FOR DATING

Archaeological materials datable by TL include ceramics, bricks and burnt clay from kilns and house walls (from burnt-down houses) and burnt stones from hearths, pits and mounds. Sherds must be at least 5 mm thick and we need about 200 g. Several small pieces making up 200 g are acceptable. Burnt flint can also be dated, but the accuracy for Nordic flint samples is usually less than can be obtained for other materials.

The best suited sediment types are eolian sediments such as loess and drift sand, but also water-lain sediments where the sedimentation has been relatively slow, e.g. marine or lacustrine sediments, are datable. Fossil soils are likewise well-suited. Certain types of glaciofluviatile sediment can also be dated, but these often have a high residual dose because the short transport and high rate of sedimentation prevent adequate exposure to sunlight.

For sandy materials, a sample of 1 kg usually contains plenty of minerals for dating. For clay-rich materials, however, it is often necessary to take several kg to obtain a sufficient amount, ca. 30 g, of the grain size used for dating.

TREATMENT OF SAMPLES

Archaeological samples are ground up in a mortar and the clay washed away, leaving only the coarse grains. All grain sizes from 0.1 to 1-2 mm are used. If the sample has been exposed to light after the excavation, the outer layer is removed before grinding.

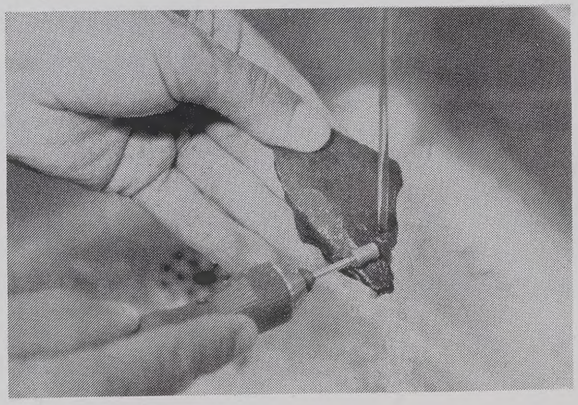


Fig. 5. The outer layer of a potsherd is removed because it has been exposed to light after the excavation and because it has received some of its beta radiation from the surrounding soil.

Sediment samples are washed to remove the clay. Clay-rich samples are treated with hydrogen peroxide or a detergent before washing. Only grains measuring 0.1-0.3 mm are used for dating.

After rinsing, quartz and alkali feldspars are separated by means of heavy liquids prepared by dissolving a heavy tungsten salt in water. The alkali feldspars are further separated into fractions with different contents of potassium. Quartz is treated with concentrated hydroflouric acid to remove plagioclase feldspar and the alkali feldspar fractions are rinsed with diluted hydrofluoric acid.

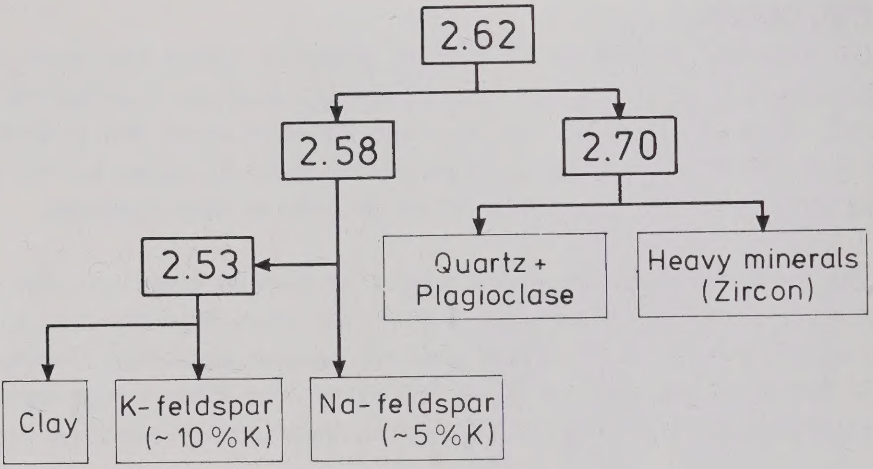


Fig. 6. Separation of minerals by means of heavy liquids prepared by dissolving a tungsten salt in water. The separation procedure yields a quartz fraction and two fractions of alkali feldspar with different concentrations of potassium. The numbers are liquid densities (g/cm^3).

DATING

The quartz and feldspar samples are heated to 500°C in an automated apparatus and the TL signal is registered. This allows a determination of the total radiation dose, the paleodose, accumulated since the zeroing event took place. By other measurements, described below, the annual dose from beta and gamma radiation is determined.

The age of the sample is then calculated from the expression:

$$\text{TL-age} = \frac{\text{Total accumulated dose}}{\text{Dose received per year}}$$

Although the formula is rather simple, the technical procedures behind it are far from simple. However, for archaeological and some sediment samples, the dating measurements can now be carried out fairly routinely.

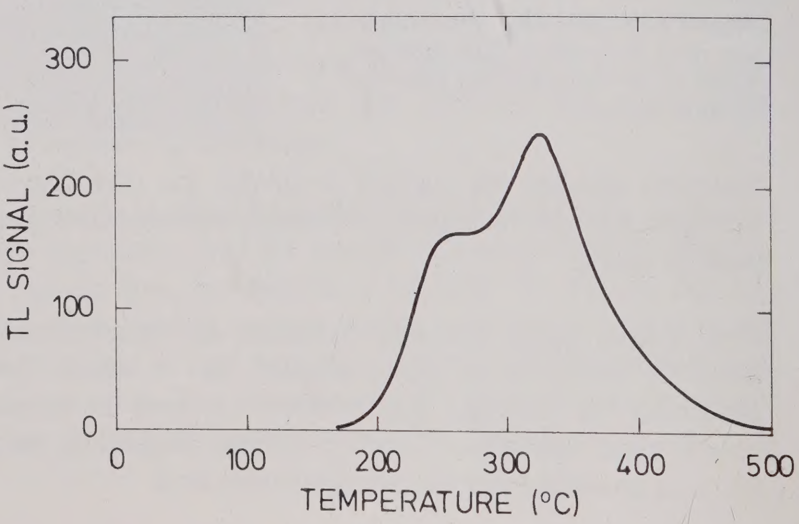


Fig. 7. TL signal from a feldspar sample plotted as a function of temperature The curve, called a glow curve, has a characteristic peak between 300 and 400°C. The area under the curve is a measure of the TL signal.

THE BACKGROUND RADIATION

When a sample is taken for dating, the natural background radiation (gamma + cosmic) to which the sample has been exposed must be determined. Normally, the measurement is carried out in the field by means of a scintillation counter; this takes only a few minutes.

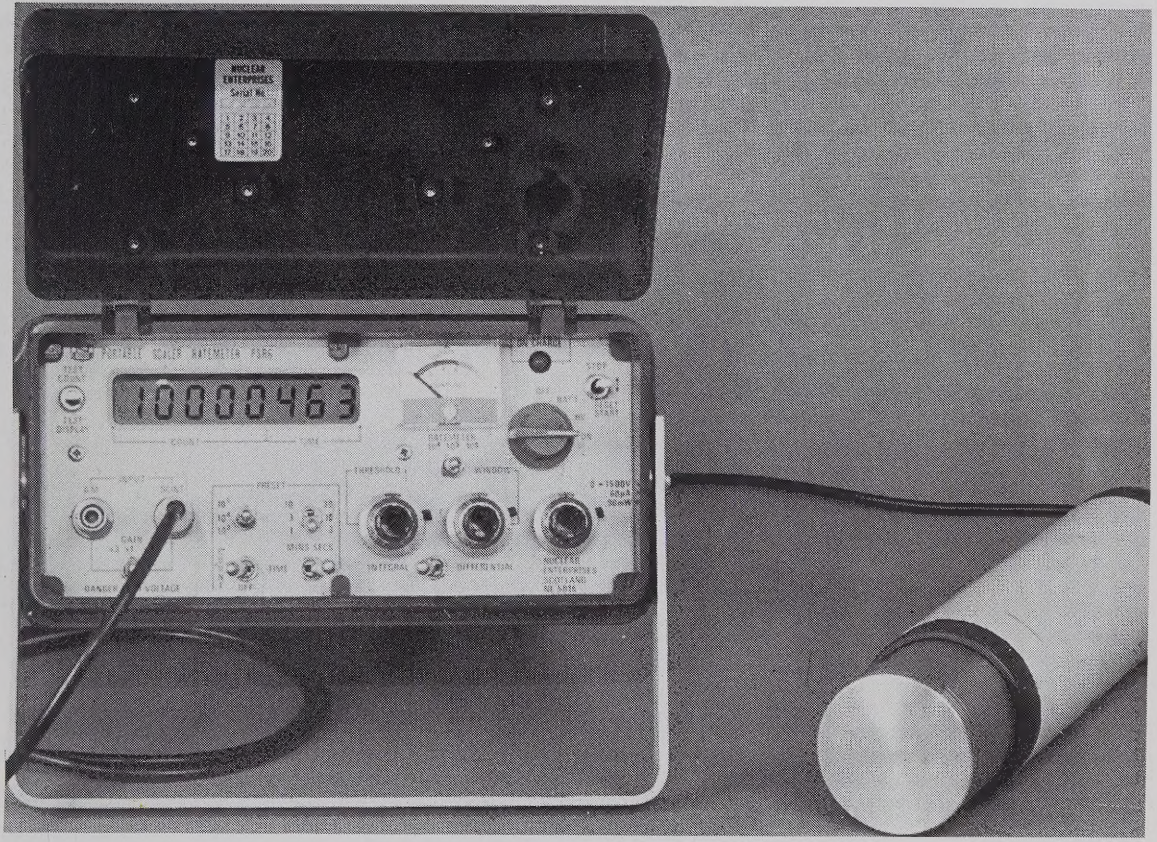


Fig. 8. Scintillation counter for measuring background radiation in the field.

In the case of archaeological samples, it is important that the excavator contacts the TL laboratory for measurement of background radiation before sample taking. On a habitation place where the soil is uniform, samples can be taken at some distance from the point where the measurement is made. For complicated structures such as pits, mounds or brick kilns, the measurement must be made at the exact spot from where the sample is taken. If measurement in the field is not possible, and soil layers are uniform, a sample of the soil (ca. 1 kg) surrounding the sample to be dated can be submitted. The gamma radiation can then be determined in the laboratory by gamma spectrometry. The soil sample can be exposed to light, but it must be sealed tightly in a bag in order to maintain its natural water content.

Measurement of the background radiation in the field is likewise preferable for sediments, because under natural conditions, water content and stratigraphy are directly taken into account. However, because sediment samples in most cases are homogeneous as far as radiation is concerned, gamma spectrometry in the laboratory is often applied directly to the sample to be dated. Measurement in the field is necessary if the strata alternates between thin layers (<30 cm) of sandy and clay-rich deposits, because the gamma radiation has a range of about 30 cm.

BETA RADIATION

Because the range of beta radiation in soil is only about 2 mm, only the radiation from the sample to be dated is of interest. The outer layer of a sherd will have received some of its beta dose from the surroundings, but, as mentioned above, this layer is removed before the dating. The beta radiation received by a grain results from both external and internal sources. The external part originates from uranium, thorium and potassium in the material surrounding the grain. In the case of a sherd, the external beta radiation originates from the clay in which the grains are embedded. The internal part, especially relevant for feldspars, originates mainly from potassium and to a smaller extent rubidium, which are constituents of the crystal lattice. Alpha radiation from uranium and thorium in the grains must also be taken into account.

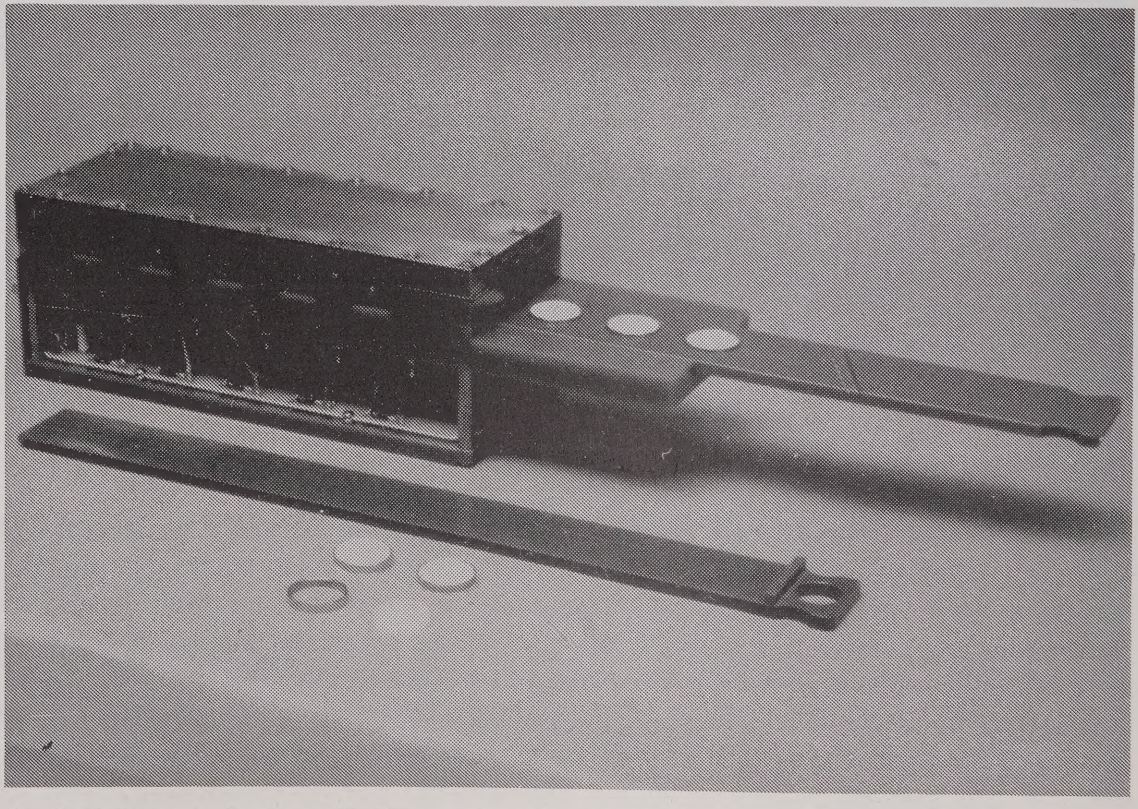


Fig. 9. Multicounter system with five counters. The counter is used for measuring beta radiation and potassium content of feldspars. The counter system was developed at Risø.

The external beta radiation is determined by 1) counting beta particles emitted by the sample by means of a multicounter system composed of five individual counters, and 2) comparing the results to a standard sample with known beta activity. The internal beta radiation in feldspar grains is calculated from a measurement of the potassium content. This measurement is carried out using the multicounter system to count beta particles from feldspar grains and comparing with a standard feldspar sample.

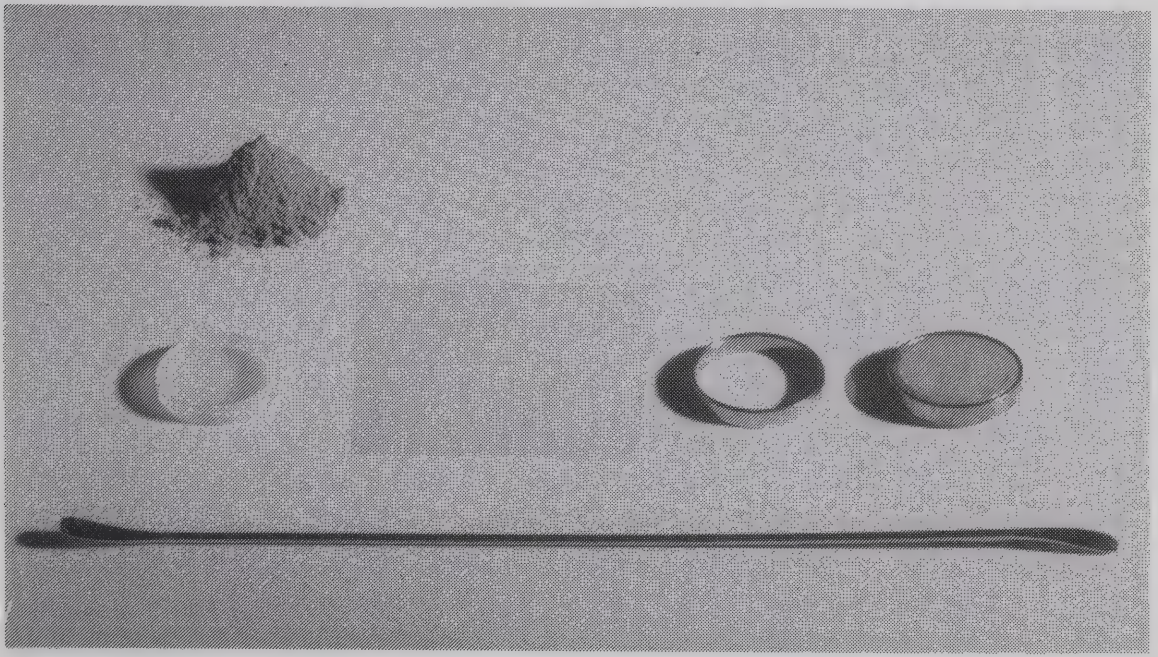


Fig. 10. Preparation of samples for beta particle counting in the multicounter system.

TIME SPAN

TL dating can be applied over a time span ranging from the present to approximately 500,000 years. The limit for quartz is about 70,000 years and is set by saturation of the TL signal, i.e. all electron traps are filled. For feldspars, saturation of the signal would set a limit at 1-2 million years. The practical limit of 500,000 years is set by the inability of the electron traps. to keep all electrons over such a long period. This loss of electrons is called fading. However, the interval back to 500,000 years covers all periods of archaeological interest as well as a considerable part of the Quaternary.

UNCERTAINTY

The main limiting factor in the accuracy obtainable in TL dating of archaeological samples, is the uncertainty of the measurements. This uncertainty is composed of random errors arising from the inevitable dispersion of measurement results, and systematic errors due, for example, to imprecise determination of the intensity of standard radiation sources used for laboratory irradiation of samples.

Only random errors are decisive in relative dating, where the task is often to decide whether two elements on a site are contemporary or not. This is usually possible if the age difference exceeds 3% of the average age of the two elements. In absolute dating, where also systematic errors must be taken into account, the uncertainty increases to 5-6% of the age. Expressed in years, the uncertainty varies from ca. 50 to 200 years from the Viking Age, to and including the Bronze Age. For the Stone Age, the uncertainty is about 300 years.

Three other sources of error are discussed briefly: short-term fading, variation of water content, and the effect of the radioactive fallout from Chernobyl.

Short-term fading occurs mainly in feldspars. A certain proportion of the electron traps in feldspars are unstable and lose their trapped charge in the course of a few weeks at room temperature. For samples irradiated in nature, all unstable traps are empty because of the long time span involved. When samples are irradiated in the laboratory, also the unstable traps are filled, so it is important that these are emptied again before the TL measurements; otherwise the age will be underestimated. For archaeological samples, we calculate a correction factor by storing laboratory irradiated samples at 100°C for 14 days and comparing their TL signals with those from samples measured immediately after irradiation. If this correction factor exceeds 15%, the sample is discarded. For sediment samples, the effect of short-term fading is eliminated by storing irradiated samples at 100°C for one week before measuring their TL signal.

Variation of water content. If the water content of soil and samples varies with time, the intensity of beta and gamma radiation will vary correspondingly because water absorbs part of the radiation. For archaeological samples from the Nordic region, the variation of water content poses no problem, because ceramics and stones in the ground are almost 100% saturated with water all year round. There are seasonal variations of the water content of the soil, but the effect on the gamma-ray intensity is negligible. However, for sediment samples, ignorance of the variation of water content during the long geological periods is a major source of error which, together with the difficulties arising in the determination of the residual dose, limit the accuracy of sediment dating to about 10% of the age.

The Chernobyl accident. A question often encountered is whether the fallout of radionuclides following the Chernobyl accident in April 1986 has any significance for TL dating. An increase of radioactivity in the soil would result in an overestimate of the annual dose, and thereby an underestimate of the age. The only place in the Nordic countries where an effect of this sort might be encountered is in Sweden, in the area north of Stockholm; here, radiation intensities up to 50 times the natural background were measured above the ground immediately after the accident.

Since then, the intensity has decreased considerably, but it will take about 100 years before it is no longer appreciable. However, the leakage from the surface into the ground is proceeding very slowly, and the fallout is still located in the upper 1-2 cm. The effect on samples taken at a depth exceeding 20 cm is, therefore, negligible.

If leakage into the ground continues in the following years, the fallout radiation can be distinguished from natural radiation by means of gamma spectrometry. It can be concluded, therefore, that the fallout from Chernobyl has no essential significance for TL dating.

WHAT CAN BE DATED BY TL

Since its establishment, the Nordic TL laboratory has dated a considerable number of archaeological samples from the Nordic countries.

In Denmark, TL has been used extensively for dating the many habitation areas from the Younger Iron Age uncovered in the last few years.

Also Bronze Age samples have been dated, i.e. from Højgård near Gram and Hyllerup, Vestsjælland. In one case, sediment dating was used to solve an archaeological problem. A tent ring was uncovered at Hjemsted south of Ribe; however, the only datable material was the drift sand that covered the site. The TL date placed the tent in the Maglemose period, 7000-8000 BC, in agreement with the archaeological estimate.

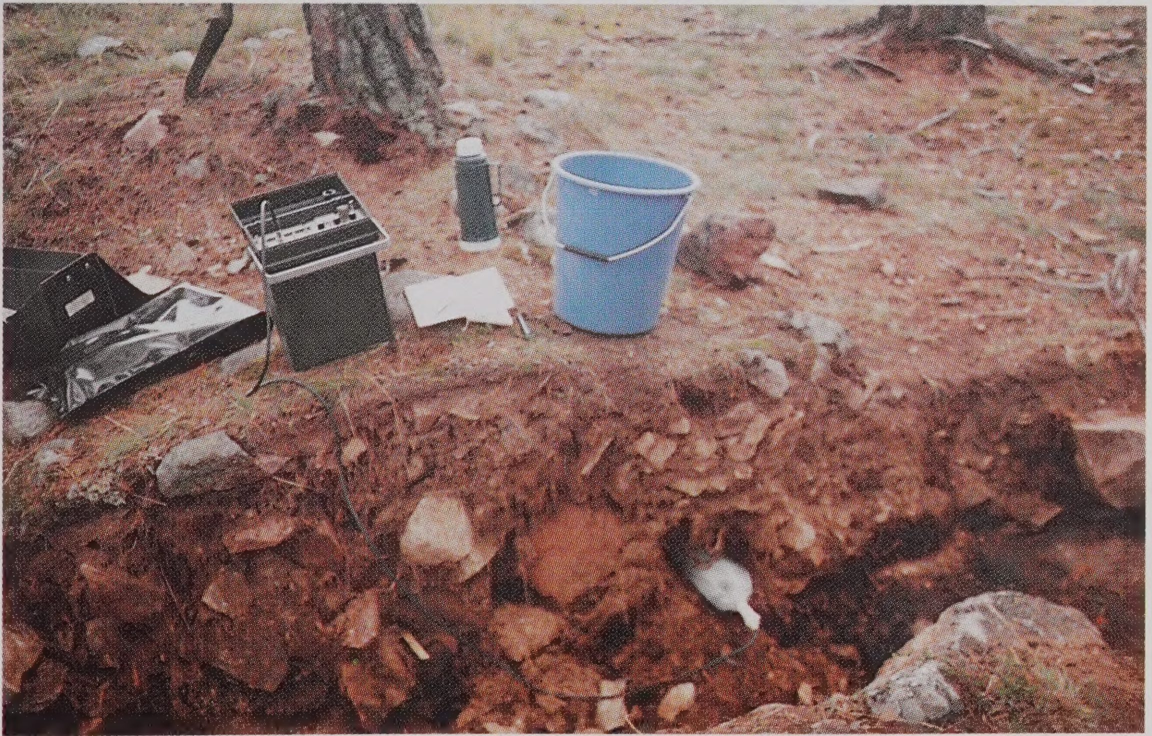


Fig. 11. Sample taking and measurement of background radiation at a hill-fort at Lingsberg, Sweden. In a structure of such complexity the radiation must be measured precisely where the sample is taken.

Major projects from Sweden include dating of burnt stones from the settlement Fosie IV, near Malmö, a series of Bronze Age mounds with burnt stones from Södermanland, and burnt stones from ancient hill-forts in the Angarnsjö area north of Stockholm.

Projects from Norway include samples from Hardangerviddan and from a mound at Etne, a mould for a church bell casting at Sola near Stavanger and samples from the Katarina hospital, Bergen.

Finnish projects include dating of bricks from a number of churches, burnt stones from a settlement at Inari and a hill-fort at Kuhmoinen.

The projects mentioned reflect a broad spectrum of archaeological tasks which have been solved by TL dating in the last few years. The work is described in a number of reports available from the laboratory.

Work on sediment dating has been concentrated on the development of suitable dating methods. In the course of this work, a number of dating tasks has also been undertaken.

Projects from Denmark include dating of ice wedges and cover sand from West Jutland, fossil soils from, for example, Oksbøl near Varde and marine deposits from Nr. Lyngby, North Jutland and Rugård, Djursland. The latter was dated to the Holstein interglacial, in agreement with the geological estimate. The fossil soil from Oksbøl was dated to the Eemian interglacial. Because the deposit had been disturbed by the ice, the dating result indicates that the ice must have advanced farther west in the Weichsel glacial period than was assumed up to now. Interglacial limnic deposits from the localities Trelde Næs and Hollerup were TL dated to Holstein and Eem, respectively, in accordance with expected ages.

Projects from Greenland include a series of samples from Kap København in North Greenland. However, these samples proved to be too old for TL dating. Good results were obtained, however, for a series of younger samples from the Thule area.

Major projects from Sweden include a number of postglacial dune sand fields from central Sweden and eolian layers at Söderåsen. Work on a series of meltwater deposits from Hven is in progress.

Norwegian projects include water-lain deposits from Finmarksviddan, Svalbard, Fjøsanger, Gudbrandsdalen, Karmøy and Jæren and drift sand layers from Sola airport. The samples from Sola are the youngest sediments dated so far. The TL dates were a little less than 2000 years, in reasonable accordance with radiocarbon dates of adjacent layers.

Projects from Finland include water-lain sediments from Inari, northern Finland and Kuhmo, eastern Finland, and dune sands from northern Finland.

CONCLUSION

The Nordic Laboratory for TL dating is engaged in dating of archaeological as well as geological samples. Its dating capacity is around 200 samples per year. Dating of archaeological samples is carried out almost on a routine basis, whereas sediment dating is still at the experimental stage. However, very promising results obtained for a range of sediments indicate that it will be possible to date at least some sediment types fairly routinely before long.

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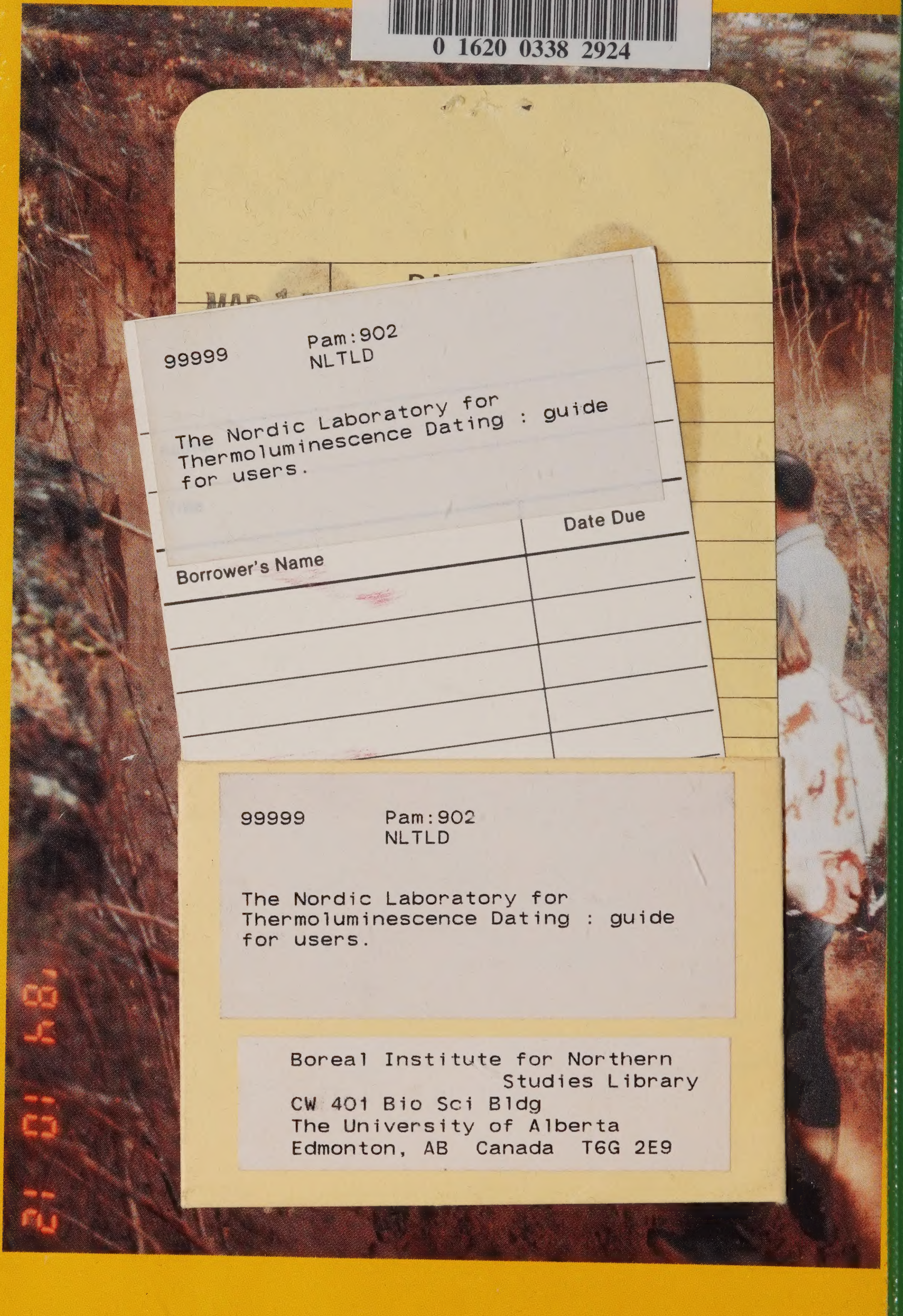

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